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LACQUERING OF SAMPLING TUBES FOR
PROTECTION AGAINST CORROSION

Army Engineer Waterways Experiment Station
Vicksburg, Mississippi

June 1959

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U. S. Army Engineer Waterways Experiment Station
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PREFACE

The U. S. Army Engineer Waterways Experiment Station, CE, was authorized to undertake the study reported herein by 2d indorsement dated 10 April 1957 from the Office, Chief of Engineers, to letter dated 19 December 1956, subject: "Project Plan for Proposed CWI Project - FY 1958." The study was performed under CWI 517, "Lacquering of Sample Tubes," of the Corps of Engineers Civil Works Investigations program, during the period 1957-1959.

Assistance in the selection and preparation of suitable paints was given by the Paint Laboratory, U. S. Army Engineer District, Rock Island.

Personnel of the Soils Division, Waterways Experiment Station, who were connected with the study were Messrs. W. J. Turnbull, W. G. Shockley, T. B. Goode, and A. L. Mathews. This report was prepared by Mr. Mathews.

Col. A. P. Rollins, Jr., CE, and Col. Edmund H. Lang, CE, were Directors of the Waterways Experiment Station during the period of the investigation and the preparation of this report. Mr. J. B. Tiffany was Technical Director.

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SUMMARY

The purposes of this investigation were: (a) to obtain or develop a lacquer, paint, or other coating for steel soil-sampling tubes which would resist abrasion, have low frictional resistance during the sampling drive, and protect the soil-sampling tubes from corrosion during storage; and (b) to develop an economical method of applying the coating to the sampling tubes.

Bending and abrasion tests, and frictional resistance tests were performed on steel test panels coated with fifteen types of coating materials. Using conventional sampling methods, fourteen undisturbed soil samples were taken from two borings in ten sampling tubes coated with the five most promising coatings as indicated by the tests on the panels, in two sampling tubes coated with the clear lacquer previously used by WES, and in two uncoated tubes. The test panels and the sampling tubes containing the samples were placed in a humid sample-storage room and periodically inspected to determine the resistance of the coatings to corrosion during storage.

Three of the coatings tested, two epoxy resins and one varnish, showed good abrasion and corrosion resistance and are considered superior to the coating previously used. Each of these coatings can be applied economically to steel soil-sampling tubes by dipping and air-drying. The resins have a lower coefficient of friction than the varnish, and one of the resins gave slightly better corrosion protection than the other. This epoxy resin is considered the best of the coatings tested and is recommended for use on steel soil-sampling tubes.

A dipping tank for coating the sampling tubes was developed and is described.

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LACQUERING OF SAMPLING TUBES FOR PROTECTION AGAINST CORROSION

PART I: INTRODUCTION

Background

1. Soil sampling and storage of soil samples in metal sampling tubes are integral parts of the soils foundation exploration work conducted by the Corps of Engineers. Friction between the soil and the walls of metal sampling tubes during sampling operations disturbs the sample structure to some degree. Furthermore, when metal sampling tubes containing certain soils are stored for any length of time, corrosion occurs on both the inside and outside walls of the tubes. Corrosion on the outside walls is of little consequence unless deep pitting occurs. However, corrosion on the inside walls is of considerable concern, as it may result in (a) chemical changes in the samples, (b) damage to the structure of the samples, and (c) increased friction between the samples and tubes to the extent that samples cannot be ejected from the tubes without serious damage to their structure.

2. At the time of this investigation, common practice for protecting metal sampling tubes from corrosion was to dip the tubes in clear hard-lacquer thinned with lacquer thinner. This lacquer provides a protective coating that permits storage of fine-grained soils in the tubes for periods of 18 to 24 months without detrimental effects. However, abrasive or coarse-grained soils scratch through the lacquer coating, and successful storage for reasonable periods of time has not been possible. The use of a tougher, more friction-free lacquer or other coating would result in decreased sample disturbance and increased corrosion resistance.

Purpose and Scope of Investigation

3. The purposes of the investigation were to:
- a. Develop, or obtain from commercial sources, a lacquer, paint, or other coating for steel soil-sampling tubes which would resist abrasion, produce little friction during the sampling drive, and protect the sampling tubes from corrosion during storage.

- b. Develop an economical method of applying the coating to sampling tubes.

4. The investigation was conducted in six stages. The scope of stage was as follows:

- a. First stage. The first stage consisted of a literature search, a canvass of coating manufacturers, and communication with the Paint Laboratory, U. S. Army Engineer District, Rock Island, to determine the availability of suitable coatings.
- b. Second stage. The second stage consisted of the selection and procurement of coatings for testing. Twelve commercial coatings (including the hard-lacquer currently used to protect sampling tubes), and seven coatings formulated and sold by the Paint Laboratory were selected and procured in sufficient quantities for the tests. The major factors considered in the selection of the coatings were: (a) "pot life" (length of time after mixing coating could be used), and (b) ease of application to steel sampling tubes.
- c. Third stage. The third stage consisted of applying the coatings to small steel test panels and subjecting the coated panels to abrasion, friction, and corrosion tests.
- d. Fourth stage. The fourth stage consisted of selecting the most promising coatings indicated by the stage-three tests and applying these coatings to steel soil-sampling tubes to be used for obtaining undisturbed soil samples in the field.
- e. Fifth stage. The fifth stage consisted of obtaining undisturbed samples of coarse-grained soils (fine sand) in the field in the coated tubes, and in uncoated tubes to be used as control specimens. Resistance of the coatings to abrasion was observed during the sampling operations.
- f. Sixth stage. The sixth stage consisted of storing the sampling tubes in a humid room for corrosion testing. The tubes have been observed for 2-1/2 months to date, and photographs taken at irregular intervals to record the progress of corrosion.

5. This report presents the essential information and results obtained in the six stages of the investigation. Because of the short time that the tubes have been in storage, it is planned to issue supplemental data at a later date if the corrosion resistance of the coatings changes significantly.

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PART 11: THE INVESTIGATION AND RESULTS

Review of Literature and Canvass of Manufacturers

6. A search of available literature and commercial pamphlets, and a canvass of thirty manufacturers of paint, rubber, and synthetic coatings were made to determine the availability of commercial products that might be suitable for coating steel coil-sampling tubes. Also, the Rock Island District Paint Laboratory was asked for information on coatings which they could formulate that might be suitable for steel sampling tubes.

7. The search, and communications from manufacturers indicated that coatings which require baking after application generally are harder and more durable than those which are simply air-dried, but the time and cost involved in the process make their use prohibitive for coating sampling tubes. Therefore, only coatings that can be air-dried were selected for testing.

Preliminary Tests Using Coated Steel Panels

Preparation of panels

8. Nineteen coating products, twelve from commercial manufacturers (including the hard-lacquer currently used) and seven from the Paint Laboratory, were obtained for testing. These products included rubber compounds, epoxy resins, silicone modified resin, vinyl resin, enamel, varnish, and lacquers, and are listed in tables 1 and 2. Duplicate 1-1/2- by 1-1/2-in. test panels of 16-gage standard cold-rolled steel were coated with these products for use in abrasion and adhesion tests. After these tests, the most satisfactory coatings were applied to a set of 3- by 6-in. panels of the same steel for use in friction tests. From the results of these preliminary tests, the coatings having the most desirable characteristics were to be selected and used to coat sampling tubes which would then be subjected to more severe tests.

9. The commercial coatings were applied by dipping the panels in the coating products which had been thinned as shown in table 1 to give a coat thickness of about 1 to 2 mils per dipping. These coatings were each

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applied to two panels which had been cleaned thoroughly in a solvent to remove the protective grease coat from the steel. In some cases one coat was applied, and in others two coats were applied to determine if a second coat would be beneficial. The coatings supplied by the Paint Laboratory were mixed and applied to the panels by the Paint Laboratory. These coatings were each applied, by dipping, to two sets of panels, one of which had been cleaned as described above, while the other had been cleaned with phosphate (pickled) after being degreased. Only one of the panels received two coats (see table 2).

10. The pot life of the coatings that were used in these tests were screened by the manufacturers, the Paint Laboratory, and the Waterways Experiment Station prior to their use in this study, and only those showing an acceptable pot life were used.

Bonding, abrasion, and corrosion tests

11. The two 1-1/2- by 1-1/2-in. coated steel test panels (each designated by the same panel number) were tested for comparative bonding and abrasion properties. The test consisted of sliding the coated specimens, loaded to 1.6 ton per sq ft of contact area, for a distance of 10 in. (length of drive on most drill rigs) over compacted, wet, fine, and coarse sand. Two test panels coated with the clear hard-lacquer (test panel 1, table 1) currently used on sampling tubes were used as control specimens for comparison. The specimens were examined visually to determine the extent of abrasion of the coatings and the approximate area of metal exposed during the tests. The results of the tests on each set are shown grouped together in tables 1 and 2.

12. After the abrasion tests the panels listed in table 1, and the uncoated panel were placed in a humid room and exposed to maximum humidity conditions for about 10 months. These panels were checked at irregular intervals to determine the corrosion resistance and durability of the coatings. In addition, the two Paint Laboratory panels which had performed satisfactorily in the previous tests (D-1 and D-3) were stored in the humid room for 2-1/2 months. The conditions of the panels after various periods of storage are shown in table 3.

13. The bonding and abrasion tests and the corrosion tests indicate that three of the coatings received from manufacturers and two of the

Laboratory coatings gave much more promising results than any of the other coatings tested. These materials were as follows:

<u>Test Panel No.</u>	<u>Coating</u>
6	Resin, epoxy, clear, catalyzed
7	Resin, epoxy, clear, catalyzed
10	Varnish, clear, catalyzed
D-1	Vinyl, aluminum
D-3	Vinyl, iron oxide

Friction resistance tests

14. Friction resistance tests were performed on one each 3- by 6-in. panel coated with the five materials listed above to determine the coefficient of friction between the coated panels and the fine, angular sand used in the bonding and abrasion tests. One panel coated with the clear hard-lacquer previously used on sampling tubes and one uncoated panel were also tested for frictional resistance for comparison. The results of these tests follow:

<u>Test Panel No.</u>	<u>Coating</u>	<u>Coefficient of Friction</u>
0	None	0.265
1	Clear hard-lacquer	0.310
6	Resin, epoxy, clear, catalyzed	0.290
7	Resin, epoxy, clear, catalyzed	0.290
10	Varnish, clear, catalyzed	0.375
D-1	Vinyl, aluminum	0.275
D-3	Vinyl, iron oxide	0.315

Tests of Coated Sampling Tubes

15. Pairs of 3-in. ID, 3-1/8-in. OD, steel soil-sampling tubes were cleaned thoroughly in a lacquer thinner bath and coated with materials identical with those used for the friction resistance test. The coatings were applied by dipping in a tank similar to that for 3-in. tubes shown in plate 1. These tubes, together with a pair of uncoated tubes, were then used to obtain undisturbed samples from two borings. One of each pair of tubes was used in each boring, with the order of the tubes reversed with respect to depth to minimize the effect of depth and soil type.

16. The samples were taken from 41- to 65-ft depths in uncased borings in fine, angular sand with a fixed piston sampler; the conventional drilling and method employed for obtaining undisturbed samples of sand from

17. The sampling tubes were removed from the humid room after 2-1/2 months, examined visually for corrosion, and photographed in black and white and in color. A 3-in.-long piece was then cut off the bottom of each tube and split lengthwise. The sample was removed from those pieces down to that portion affected by corrosion, and the inside walls of the pieces of tube were examined and were also photographed in black and white and in color. The tubes then were returned to the humid room for continuation of the corrosion tests. The condition of the sampling tubes as determined from the visual inspection is given in table 4. The photographs are on file at WES.

18. In view of the short period (2-1/2 months) during which the tubes were subjected to corrosion tests in the humid room before this report was prepared, additional visual inspections and color photographs, for comparison with the first photographs, will be made of the outside and inside of the sampling tubes at 6-month intervals to determine the extent and effects of corrosion after further exposure in the humid room. Supplemental data will be published at a later date in the event that the additional storage time indicates that the corrosion resistance of the respective coatings over longer periods of storage is significantly different from that for a 2-1/2-month period as shown in table 4.

¹ M. J. Thompson, *Journal of the American Chemical Society*, **80**, 1416 (1958); *ibid.*, **81**, 1416 (1959).

PART III: SUMMARY OF RESULTS

19. The literature search and canvass of various manufacturers indicated that coatings which require baking after application generally are harder and more durable, but the excessive time and cost involved would make their use prohibitive for coating sampling tubes. Therefore, only coatings that could be air-dried were considered for the tests.

20. Coatings applied to panels that had been solvent-cleaned only withstood the abrasion tests as well as those applied to panels that had been solvent-cleaned or phosphate-cleaned (pickled). Consequently, panels and sampling tubes that had been solvent-cleaned only were used in subsequent tests.

21. Two-coat application of coatings was investigated in the preliminary tests, but this procedure was discarded because of the extra time and cost involved and the lack of improvement in the qualities desired in the coatings.

22. The pot life of coating materials was considered, as a short pot life would entail considerable waste when coating a small number of sampling tubes. Some coatings with a relatively short pot life, such as the catalyzed coatings (panels 6, 7, and 10), were the most abrasion resistant and were included in the final tests, as the good qualities of the coatings obtained justified the additional cost resulting from the unavoidable wastage of coating material.

23. The friction tests showed that test panel D-1 had the lowest coefficient of friction, with the coefficient of friction of test panels 6 and 7 only slightly higher, and only 9.5 per cent greater than that of the test panel with no coating. The other two test panels, 10 and D-3, had coefficients of friction of 41.5 per cent and 13.9 per cent, respectively, greater than the uncoated test panel.

24. Examination of the sampling tubes after storage in the humid room for 2-1/2 months indicated that tube 6 from boring 1 showed the least corrosion, and tubes 6, 7, and 10 from boring 2 showed about the same condition. A considerable amount of the coatings on all tubes was abraded from the outside of the tubes during the sampling drive. The coating on tubes 6, 7, and 10, although apparently abraded to the bare metal on some

areas, gave good protection against corrosion, indicating that the entire surface was still covered by the coating even though thinly over some areas. Tests indicate that the two catalyzed epoxy resin coatings used on test panels 6 and 7 had the same low coefficient of friction and were equally corrosion resistant. These coatings were equally corrosion resistant on sampling tubes 6 and 7 from boring 2, but tube 6 from boring 1 showed more corrosion resistance than tube 7 from boring 1.

PART IV: CONCLUSIONS AND RECOMMENDATIONS

Coatings

25. The clear epoxy resin coating used on tube 6 gave as good or better results than the other coatings in both preliminary and final tests; therefore, it is recommended as the best coating for soil-sampling tubes. The manufacturer recommended the use of the coating and catalyst in 1:1 proportion by volume, which gave a material of satisfactory as-mixed viscosity for dipping. The maximum pot life of the material is 8 hours in cool weather, and may be as low as 6 hours in hot summer weather. The coating will dry in a very short time but will not be fully cured and ready for field use in less than 7 days.

26. Before the coating is applied, the tubes should be thoroughly cleaned with a suitable solvent, such as a 1:1 mixture of mineral spirits and xylol, and wiped dry with a clean cloth. The coating should be applied by dipping in a tank similar to the one described below, and the coated tubes allowed to dry in a vertical position with the cutting edge up. (With the tube in a vertical position any excess coating will drain downward and any resulting irregularity of the coating will be at the top of the tube, which does not come in contact with the soil sample.) The sampling tubes should be withdrawn from the coating material at a constant rate of approximately 1 ft per sec, which will give a coating thickness of 0.001 to 0.0015 in. when the air temperature is about 70 F. The slower the withdrawal rate, the thicker the coating obtained.

Dipping Tanks

27. Satisfactory tanks for dipping tubes are shown in plate 1. With the inner tubing of the tank plugged at the top as shown, only a small quantity of coating material is required to fill the tank, and wastage of material remaining after the sampling tubes are coated is minimized. The screw connection at the bottom of the outer tube will permit disassembly for easy cleaning. By using tubing or pipe of the diameters shown, any lateral movement of the sampling tube during withdrawal will force the

outside of the sampling tube to touch the inside wall of the outer tubing of the tank and prevent the inside of the sampling tube from touching the inner tubing of the tank. Thus, any disturbance or marring of the coating due to contact of the sampling tube with the tank during withdrawal will be on the outside of the sampling tube, and a smooth, uniformly covered surface on the inside of the sampling tube will be assured.

Table 1

Results of Pencil and Abrasion Tests on Coatings Received From Manufacturers

Test Panel No.	Coating	Type of Primer	Type of Catalyst	Mixing Proportions		Texture of Coating	Abrasion After Test	Adhesion Performance	
				Coating	Primer				
1	Epoxy, hard, clear	Isocyanate thinner	None	2	1	0	Smooth	1/3	Poor
2	Rubber, chlorinated, gray	M.E.K.*	None	1	1	0	Smooth	2/5	Poor
3	Resin, epoxy, red	**	**	2	1	+	Smooth	1/4	Poor
4	Resin, epoxy, gray	**	**	2	1	+	Smooth	Trace	Fair
5	Resin, epoxy, clear	XC-101††	**	2	1	+	Smooth	1/3	Poor
6	Resin, epoxy, clear	None	BL-14533††	1	0	1	Smooth	None	Good
7	Resin, epoxy, clear	None	**	8	0	1	Smooth	None	Good
8	Resin, epoxidized, clear	M.E.K.	XY-23††	4	3	+	Smooth	1/3	Poor
9	Resin, ethyl, clear	M.E.K.	None	3	1	0	Irregular w/bubbles	Trace	Fair
10	Urethane, clear	Turpentine	IR-1674††	8	1	+	Smooth	None	Good
11	Isocyanate, polybutadiene, chlorinated, hard	**	None	3	2	0	Irregular	Trace	Fair
12	Coating as applied by manufacturer, clear	--	--	-	-	-	Smooth	1/4	Poor

Note: All specimens given one coat except panel 4 which had one coat applied over one coat of primer (coating 3). All panels were solvent-cleaned and unpickled except panel 12 which was pickled.

* Methyl ethyl ketone.

** Type as furnished by manufacturer, not identified.

† Proportion as furnished by manufacturer, exact proportion not determined.

†† Manufacturer's name, not identified otherwise.

Table 2
Bonding and Abrasion Tests on Coatings Prepared
by Rock Island Paint Laboratory

Test Panel Number*	Coating**	Texture of Coating	Abrasion Resistance	Remarks
D-1 P-1	Aluminum vinyl (1 coat)	Smooth Smooth	Good Good	Satisfactory Satisfactory
D-2 P-2	Aluminum vinyl (2 coats)	Smooth Smooth	Good Good	Coating too thick Coating too thick
D-3 P-3	Iron oxide vinyl (1 coat)	Smooth Smooth	Good Good	Satisfactory Satisfactory
D-4 P-4	Iron oxide vinyl (2 coats)	Smooth Smooth	Good Good	Coating too thick Coating too thick
D-5 P-5	Epoxy polyamide (1 coat)	Smooth Smooth	Fair Fair	Abrasion resistance unsatisfactory Abrasion resistance unsatisfactory
D-6 P-6	Epoxy amine (1 coat)	Smooth Smooth	Fair Fair	Abrasion resistance unsatisfactory Abrasion resistance unsatisfactory
D-7 P-7	Zinc-rich vinyl (1 coat)	Rough Rough	Good Good	Texture unsatisfactory Texture unsatisfactory
D-8 P-8	Zinc-rich phenolic (1 coat)	Rough Rough	Good Good	Texture unsatisfactory Texture unsatisfactory
D-9 P-9	Zinc-rich epoxy (1 coat)	Rough Rough	Good Good	Texture unsatisfactory Texture unsatisfactory

* "D" preceding the number denotes specimen solvent-cleaned; "P" preceding the number denotes specimen phosphoric-acid-cleaned (pickled).
 ** All specimens given same kind and number of coatings.

Table 3

Condition of Panels After Various Periods of Storage in Humid Room

Test Panel*	16 June 1958	24 November 1958	24 January 1959	9 April 1959
0**	Placed in storage	Medium rust	Heavy rust	Heavy rust
1	Placed in storage	Medium rust	Heavy rust	Heavy rust
2	Placed in storage	Light rust at abrasions	Medium rust at abrasions	Medium rust at abrasions
3	Placed in storage	Light rust at abrasions	Medium rust at abrasions	Medium rust at abrasions
4	Placed in storage	No rust	Light rust under coating	Light rust under coating
5	Placed in storage	Trace of rust under coating	Light rust under coating	Light rust under coating
6	Placed in storage	No rust	Rust coloration under coating	Rust coloration under coating
7	Placed in storage	No rust	Rust coloration under coating	Rust coloration under coating
8	Placed in storage	Trace of rust at abrasions	Light rust at abrasions	Medium rust at abrasions
9	Placed in storage	Trace of rust under coating	Light rust under coating	Medium rust under coating
10	Placed in storage	No rust	Rust coloration under coating	Rust coloration under coating
11	Placed in storage	Trace of rust under coating	Medium rust under coating	Medium rust under coating
12	Placed in storage	Medium rust	Heavy rust	Heavy rust
D-1	--	--	Placed in storage	Trace of rust at abrasions
D-3	--	--	Placed in storage	Trace of rust at abrasions

* For identification of test panels, see tables 1 and 2.

** Uncoated panel.

Table 4

Condition of Coated Sample Tubes After 2-1/2 Months,
as Determined From Visual Inspection

Sample No. *	Tube No. **	Condition of Tube	
		Outside of Tube	Inside of Tube
1-1	1	Fine rust covering 75% of area	Heavy rust penetrating sample 1/16 in.
1-2	3	Fine rust covering 40% of area	Heavy rust penetrating sample 1/16 in. on 1/5th of area
1-3	6	Trace of fine rust	Trace of fine rust
1-4	7	Trace of fine rust with spots of fine rust	Medium rust penetrating sample 1/32 to 1/16 in.
1-5	10	Trace of fine rust	Trace of rust with medium rust penetrating sample 1/32 in. 1/16 in. on 1/10th of area
1-6	D-1	Fine rust covering 40% of bare area on tube	Medium rust on bare places penetrating sample 1/32 in. on 1/20th of area
1-7	D-3	Fine rust covering 40% of bare area on tube	Medium rust on bare area penetrating sample 1/32 in. on 1/20th of area
2-1	D-3	Fine rust covering 50% of bare area on tube	Trace of medium rust with penetration of sample 1/32 in.
2-2	D-1	Fine rust covering 40% of bare area on tube	Trace of fine rust
2-3	10	Trace of fine rust	Trace of fine rust
2-4	7	Trace of fine rust	Trace of fine rust
2-5	6	Trace of fine rust	Trace of fine rust
2-6	3	Fine rust covering 40% of bare area	Medium rust penetrating sample 1/32 to 1/16 in. on 1/10th of area
2-7	1	Fine rust covering 75% of area	Heavy rust penetrating sample 1/16 in.

